LLNL-CONF-691577

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Mechanism and simulation of removal rate and surface roughness creation during optical polishing of glasses

T. Suratwala, R. Dylla-Spears, R. Steele, N. Sheen, M. Feit, R. Desjardin, L. Wong, P. Miller

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Mechanism and simulation of removal rate and surface roughness creation during optical polishing of glasses

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Session title: Mechanical Properties of Glasses V
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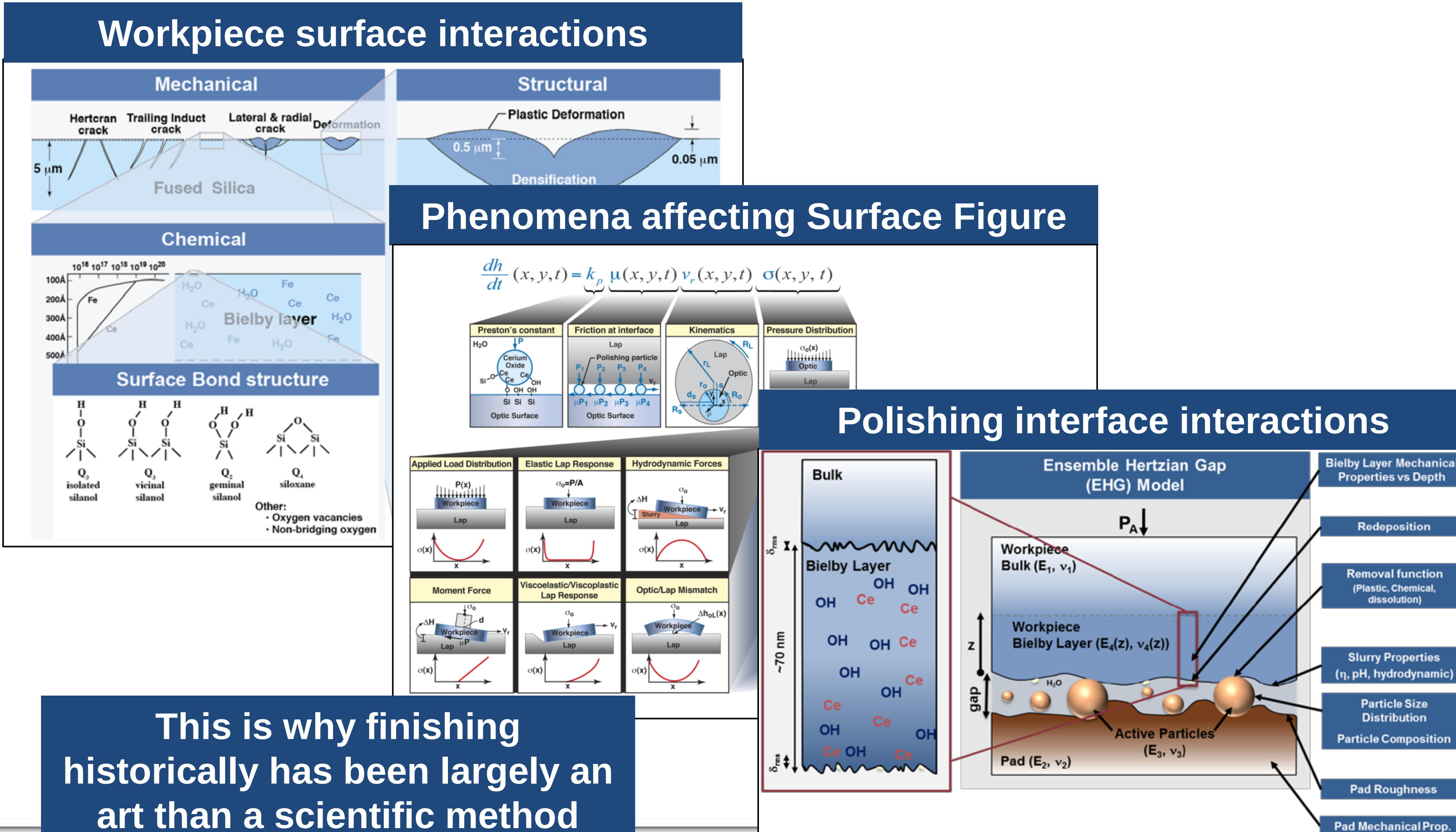
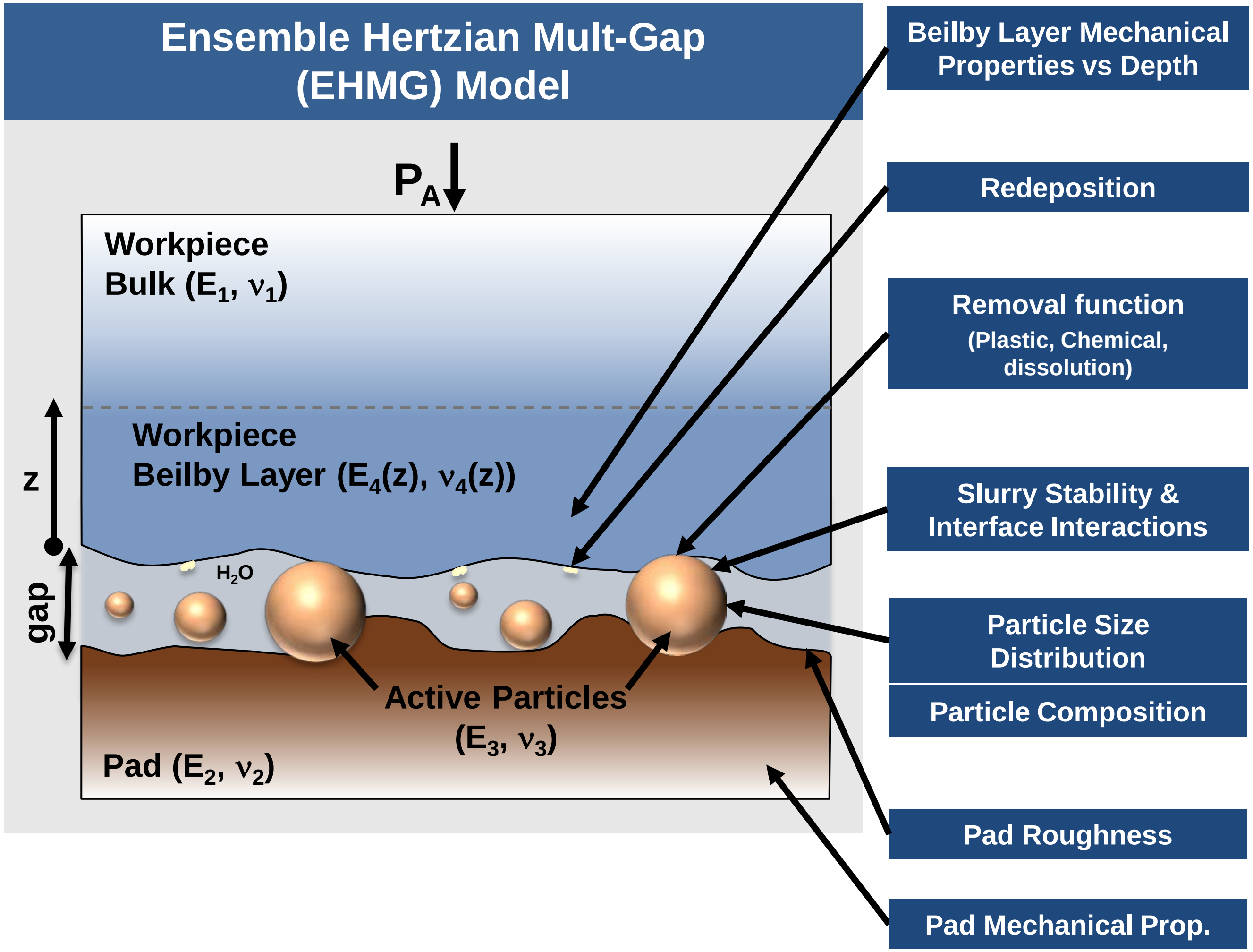
To date, the complexities of polishing has made is difficult to scientifically design, optimize a process for a given material



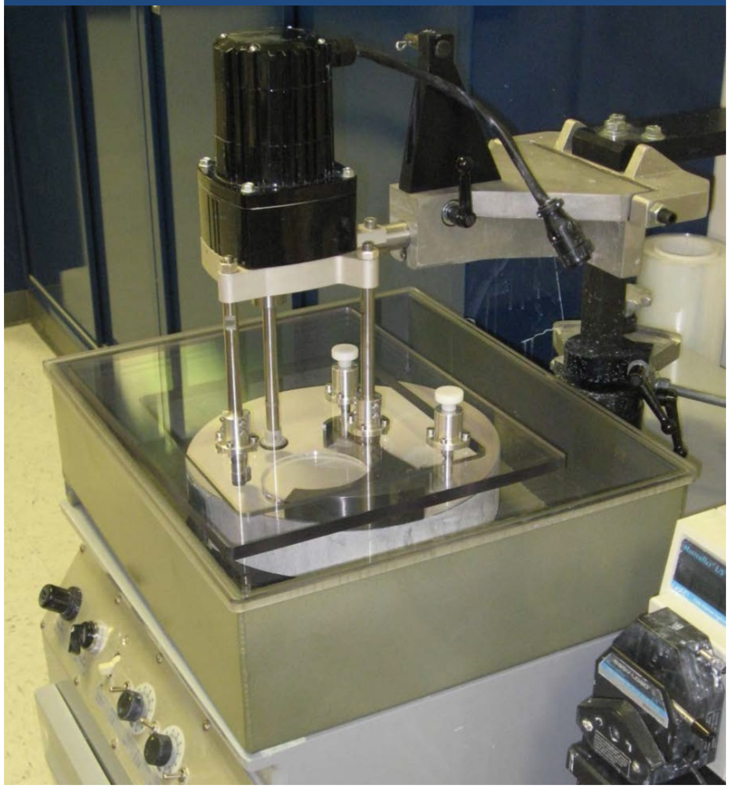
This is why finishing historically has been largely an art than a scientific method

Schematic model of the parameters that affect roughness during polishing



Polishing was conducted using the Convergent Polishing Method (ceria or silica slurry on various glasses using a polyurathane pad)

CISR0 polisher

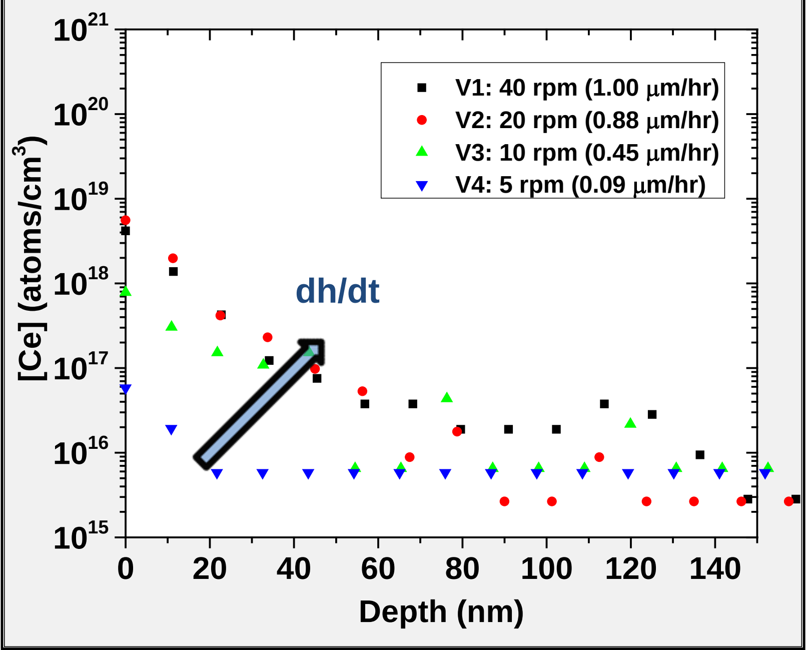


CISR1 polisher

These polishing systems offer great control over process parameters (temperature, humidity, PSD, rogue particles, pad treatment etc.) & diagnostics allowing for very controlled, repeatable polishing experiments

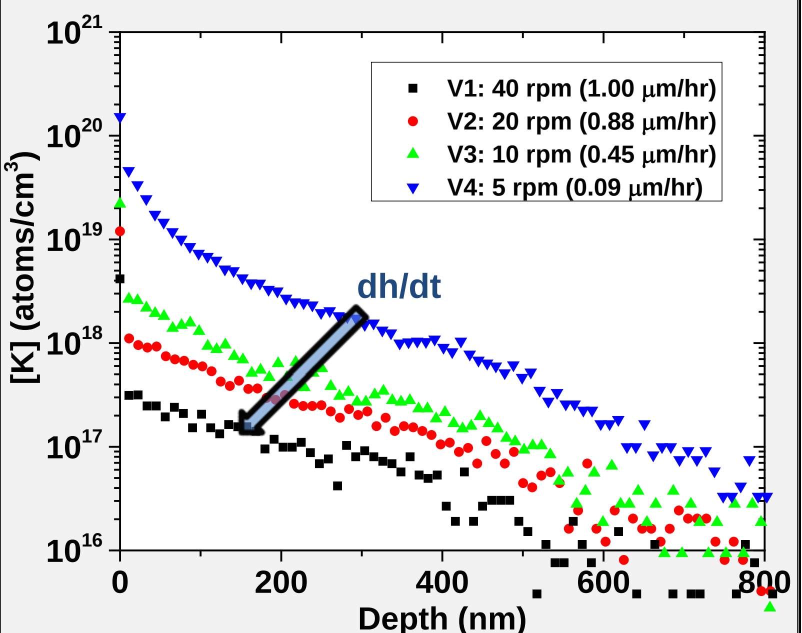
Our measurements show that Ce penetration is not due to diffusion & K penetration is consistent with diffusion

[Ce] profile on polished fused silica surface as fn of polishing velocity



Ce penetration increases with increase in removal rate; suggestive of a surface reactivity mechanism; Ce is the active component in material removal during polishing

[K] profile on polished fused silica surface as fn of polishing velocity

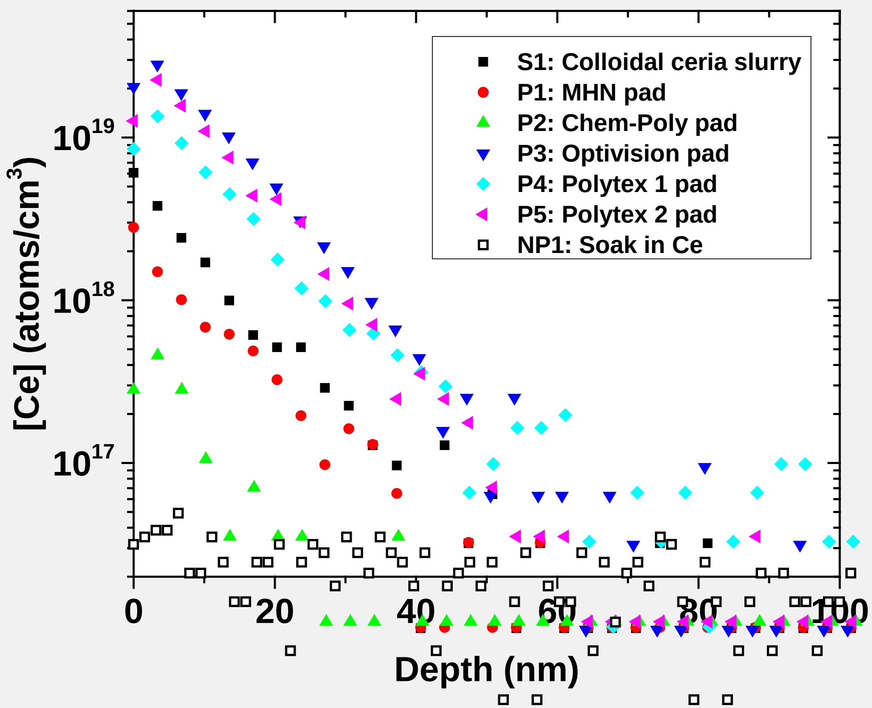


K penetration decreases with increase in removal rate; consistent with diffusion

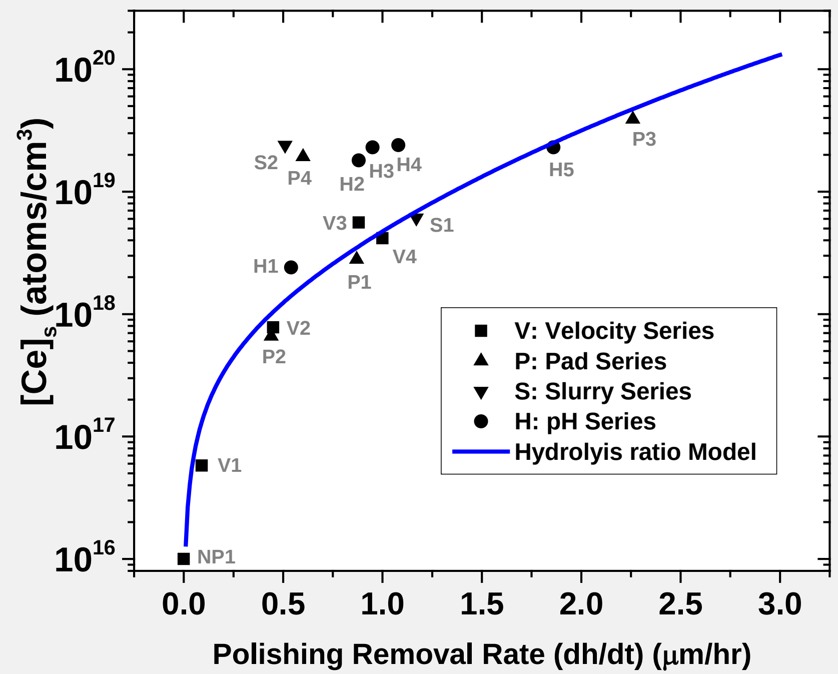
[1]Measured by SIMS (note Si 2×10^{22} atom/cm^3)

[Ce]$_s$ increases with polishing removal rate & is weakly dependent on other polishing parameters

[Ce] of polished surface layer for variety of polishing conditions



Correlation between [Ce]$_s$ and removal rate (dh/dt)



T. Suratwala et. al., *J. Am. Cer. Soc* 98(8) (2015) 2396

The penetration of Ce into silica surface during polishing is proposed to be a competition of hydrolysis reactions

Mechanism

1) Removal rate increases
2) Interface temperature increases
3) Arrhenius increase to r
4) Greater Ce surface deposition

Condensation

≡Si-OH + HO-Ce ≡ → ≡ Si-O-Ce ≡ + H_2O

Silica Hydrolysis

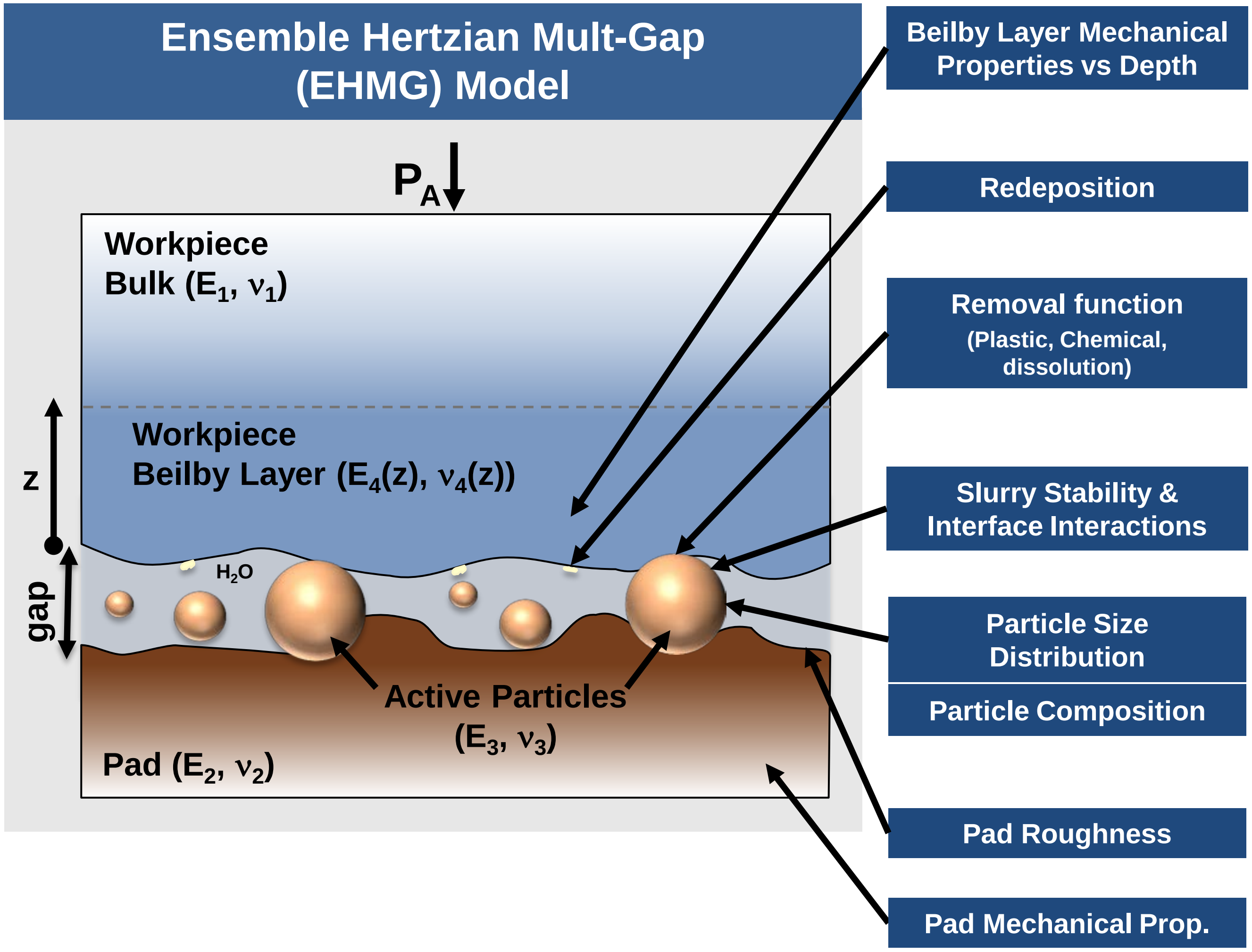
≡Si-O-Si-O-Ce-O-Ce ≡ + H_2O → ≡ Si-OH + HO-Si-O-Ce-O-Ce ≡

Ceria Hydrolysis

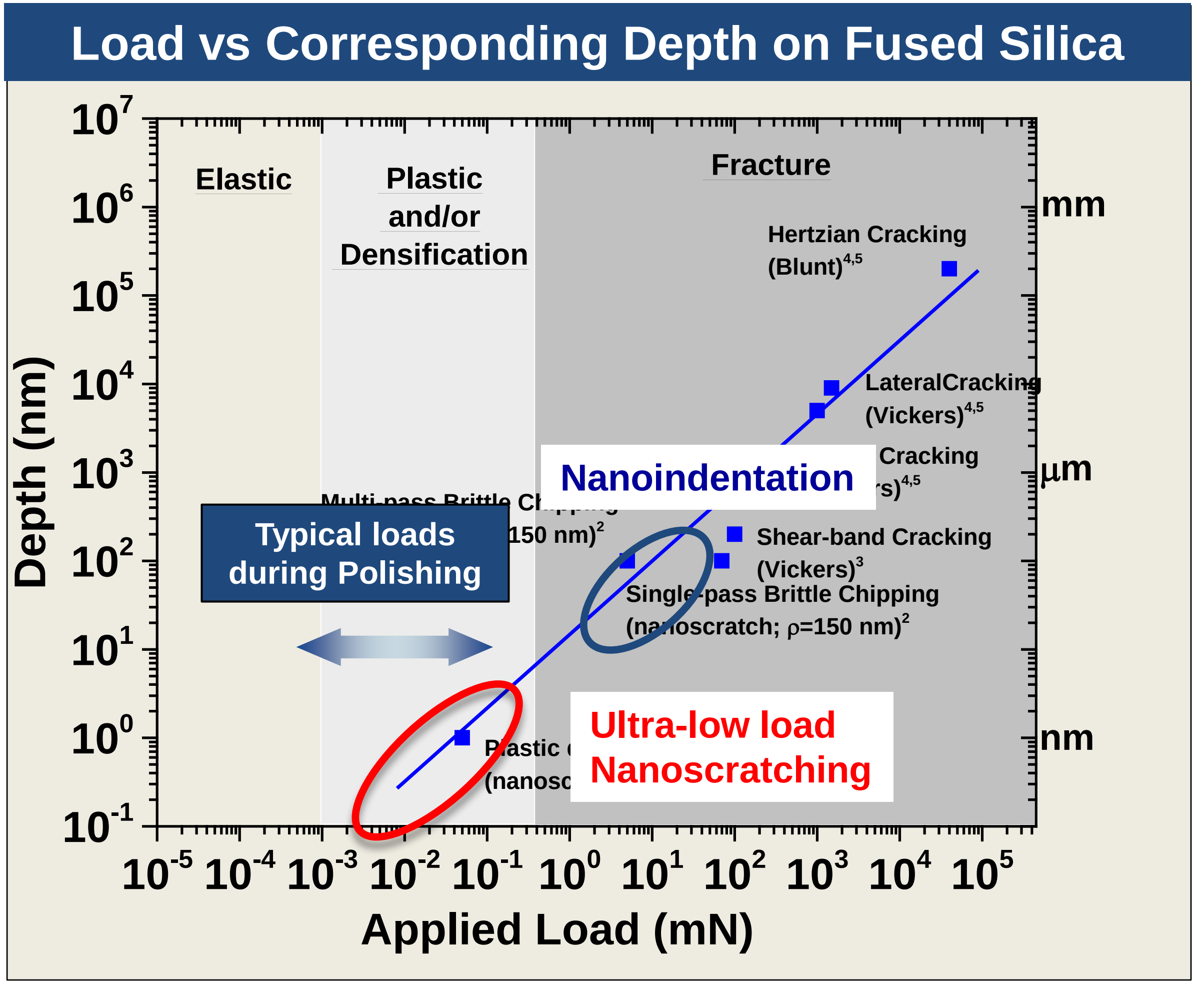
≡Si-O-Si-O-Ce-O-Ce ≡ + H_2O → ≡ Si-O-Si-O-Ce-OH + HO-Ce ≡

r =Ceria Hydrolysis rate/ Silica Hydrolysis rate

Schematic Model of the parameters that affect roughness during polishing



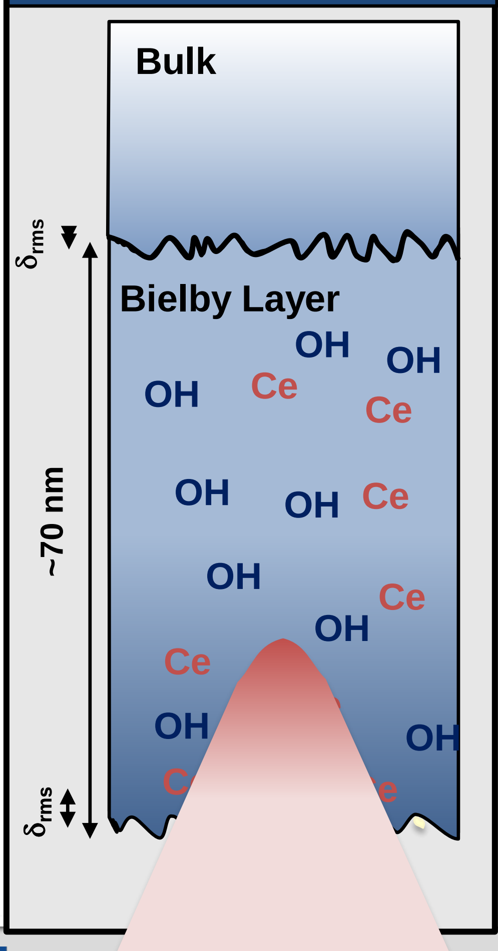
Nanoscratching at ultralow loads offers exploring mechanical properties of Bielby layer and particle removal function



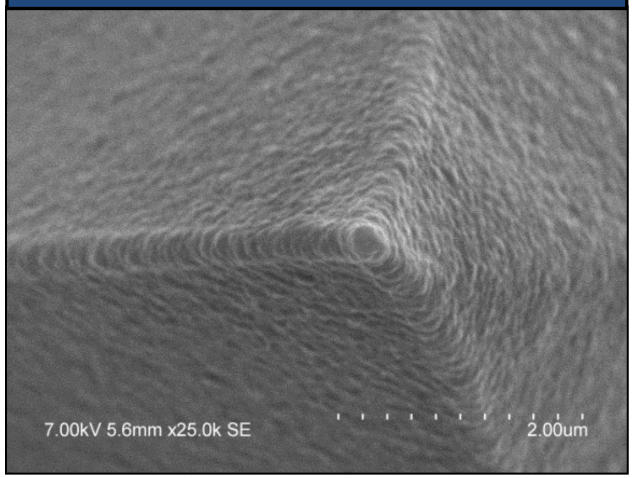
[1] This study (2014); [2]Thongoom *J. Mat. Sci* 40 (2005);[3]Miller, Optics Letters 35(16) (2006); [4]Lawn, *Fracture of Brittle Solids* (1993);[5]Suratwala, JNCS 354 (2008)

Using a stiff AFM tip, nanoscratching has proven a viable method to explore the mechanical properties of Beilby layer

Schematic of Beilby Layer



SEM of a AFM diamond tip

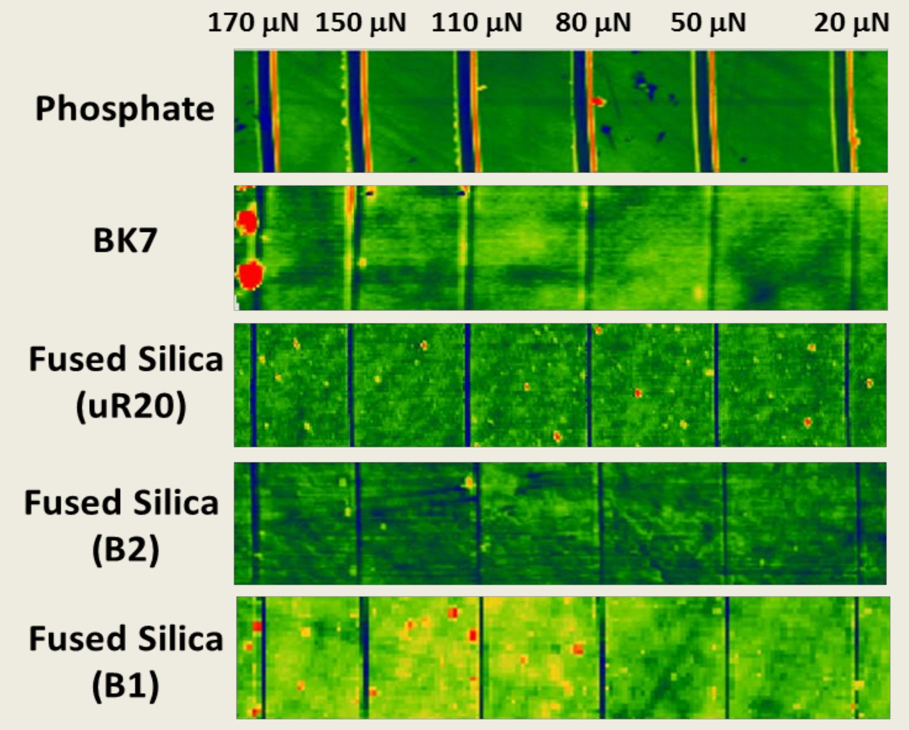


<u>Nanoscratching Method</u>

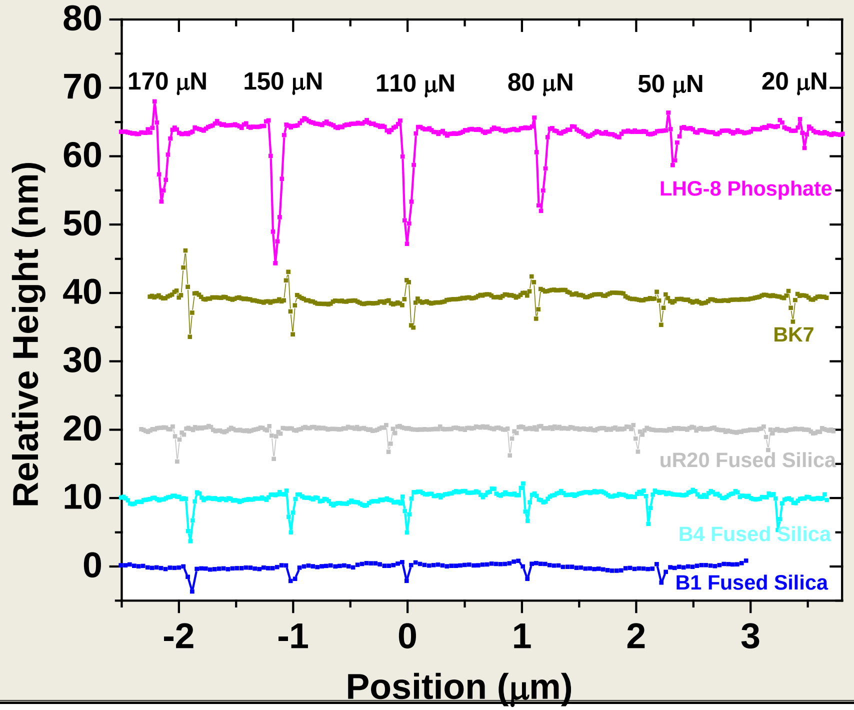
- **Standard AFM tip (Si; 0.1-1 N/m; ~10 nm radius) replaced with Stiff AFM tip (Diamond; 42 N/m; 150 nm radius)**
- **Nanoscratches created at loads 0 – 170 μN**

Fused silica and BK7 show little load dependence on permanent deformation; changes in Bielby layer of fused silica influences depth

AFM images of nanoscratches on different surfaces at various loads

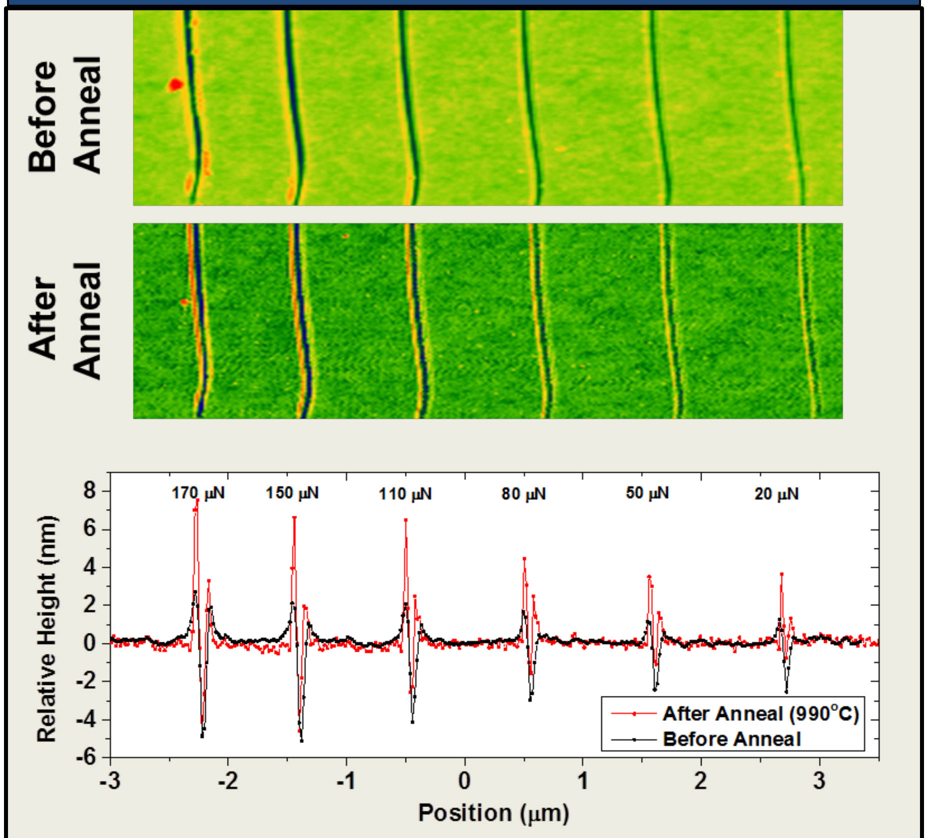


Cross-section of nanoscratches at various loads on various substrates

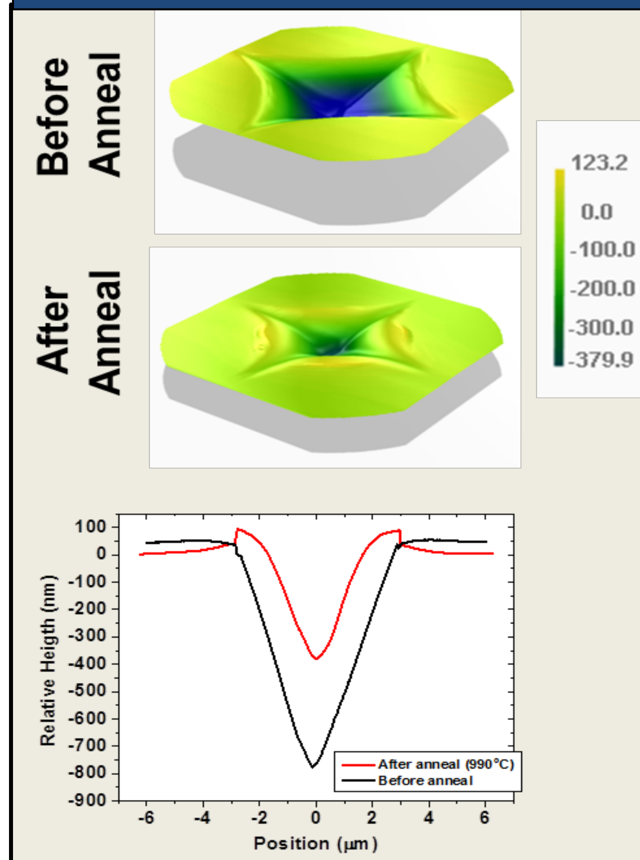


Annealing induced relaxation[1] supports that nanoscratches on fused silica are largely due to densification

Nanoscratches before & after annealing



0.5 N Vickers indentation before & after annealing

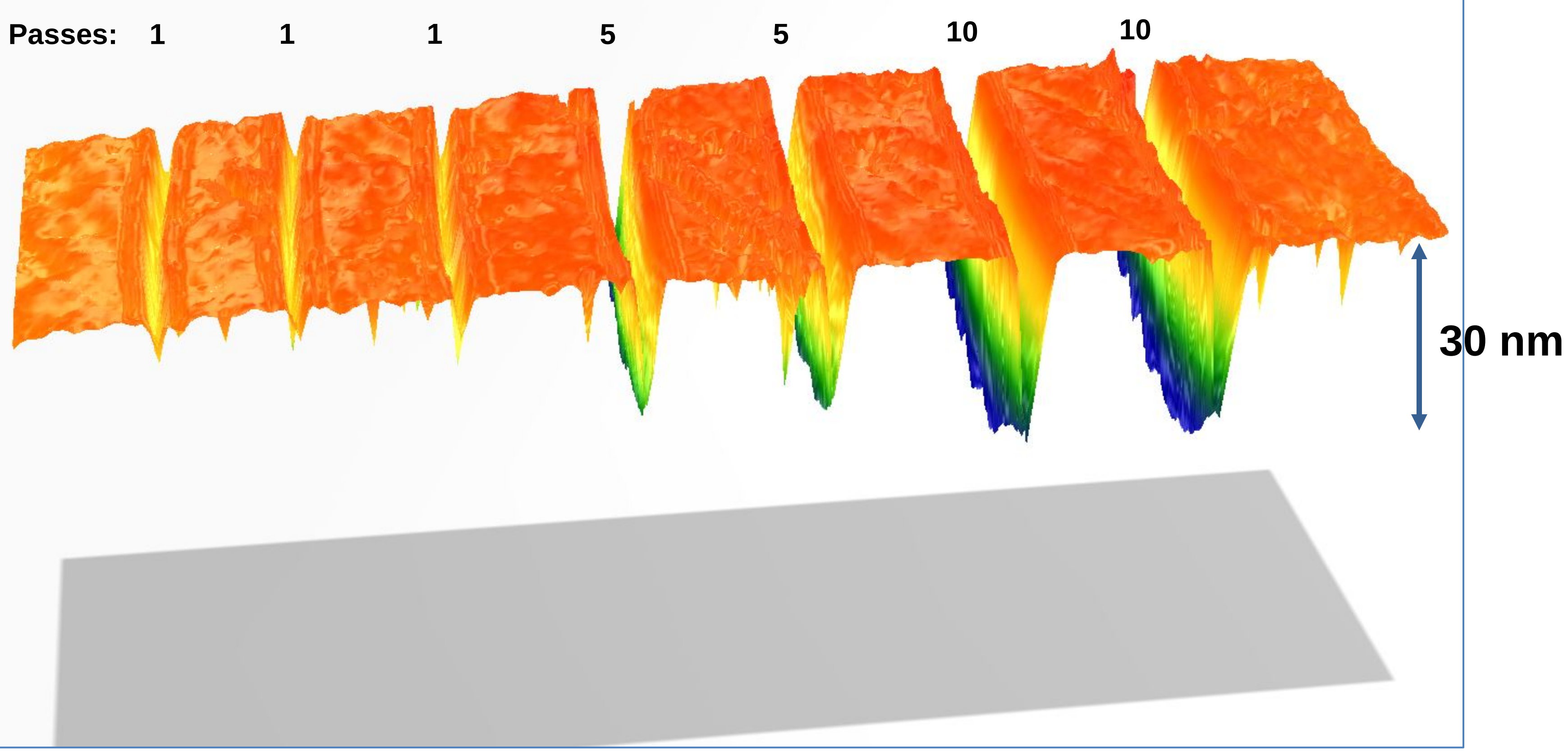


Annealing temp 0.9 Tg

[1] Using technique described by Yoshida .et. al., J. Mater. Res., Vol. 20, No. 12, Dec 2005

The removal volume was determined from multi-pass nanoscratching to account densification effects

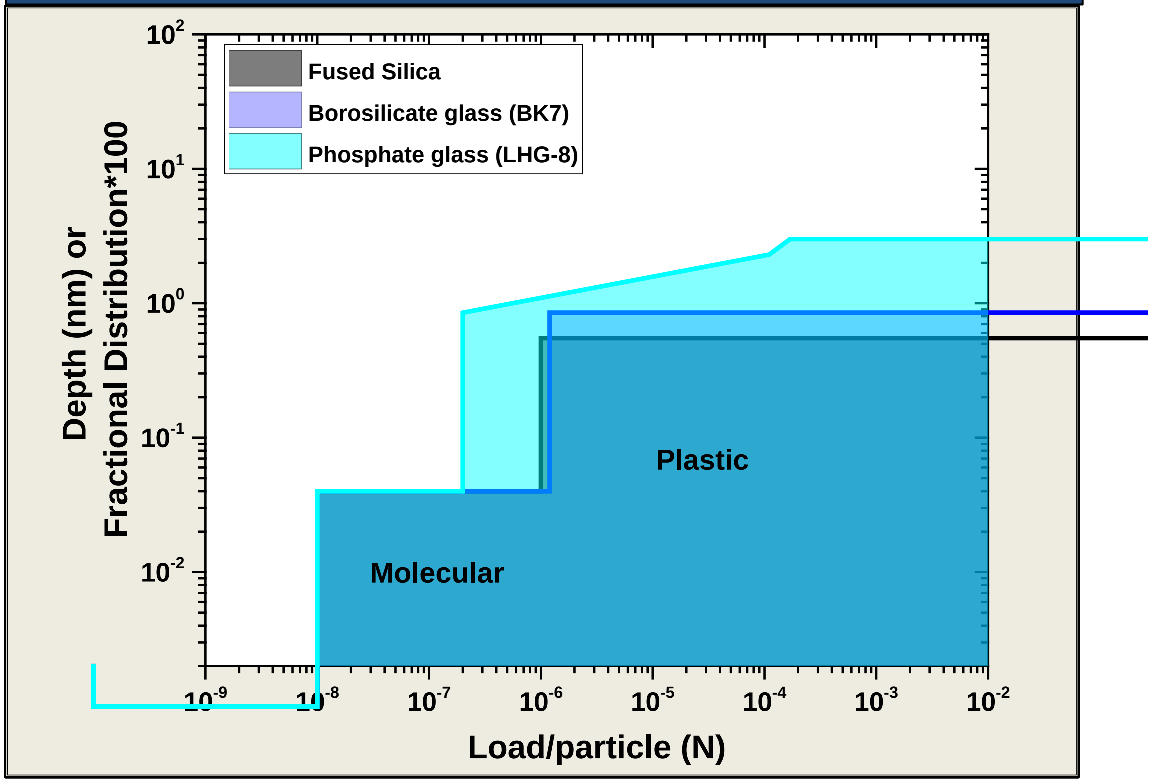
LHG-8 phosphate glass: scratches at 110 μN



N. Shen et. al., *J. Am. Cer. Soc* (2016) 1-8

A detailed description of the removal function has been determined for various glasses aiding to the prediction of roughness

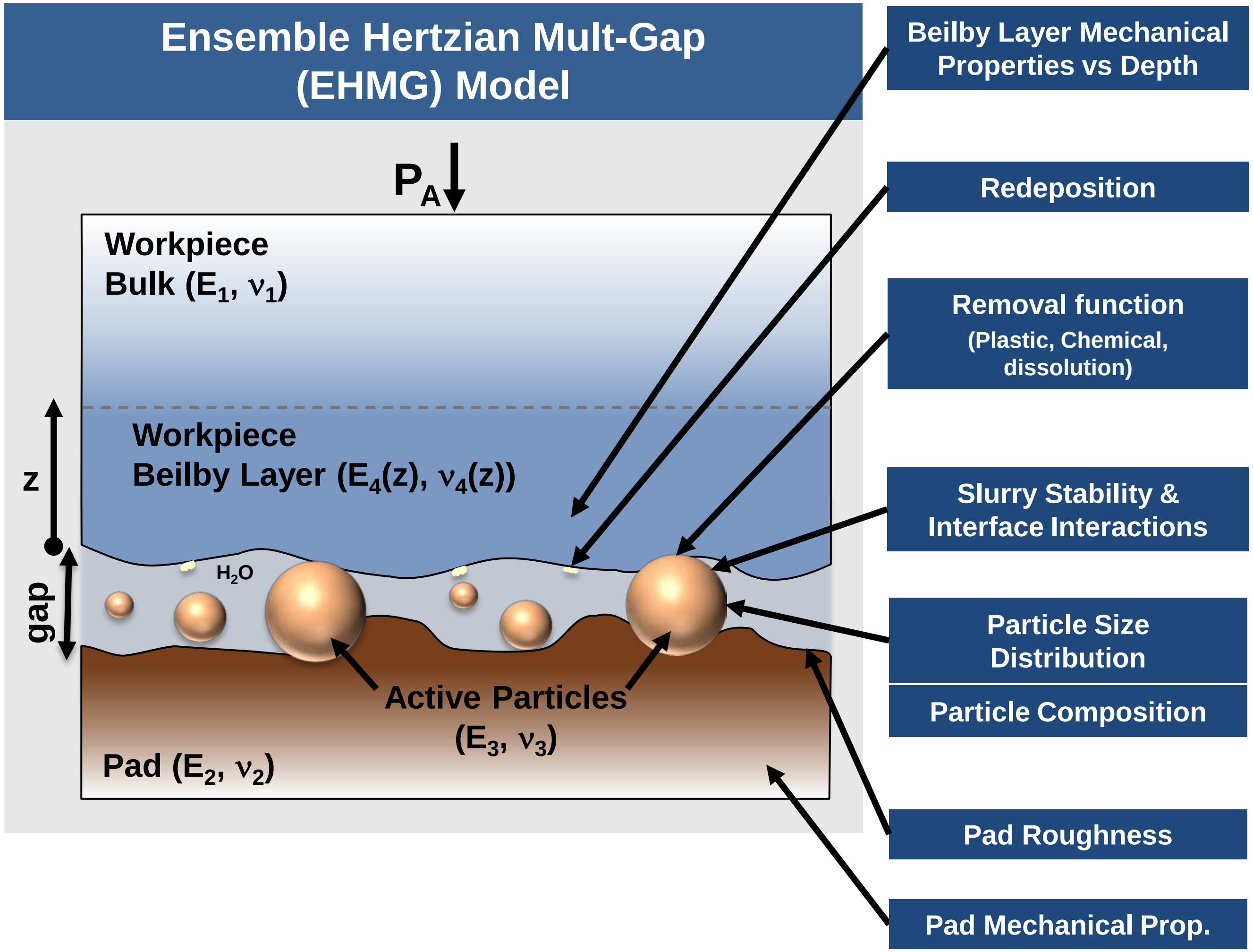
Determined removal function for single particle on various glasses



N. Shen et. al., *J. Am. Cer. Soc* (2016) 1-8

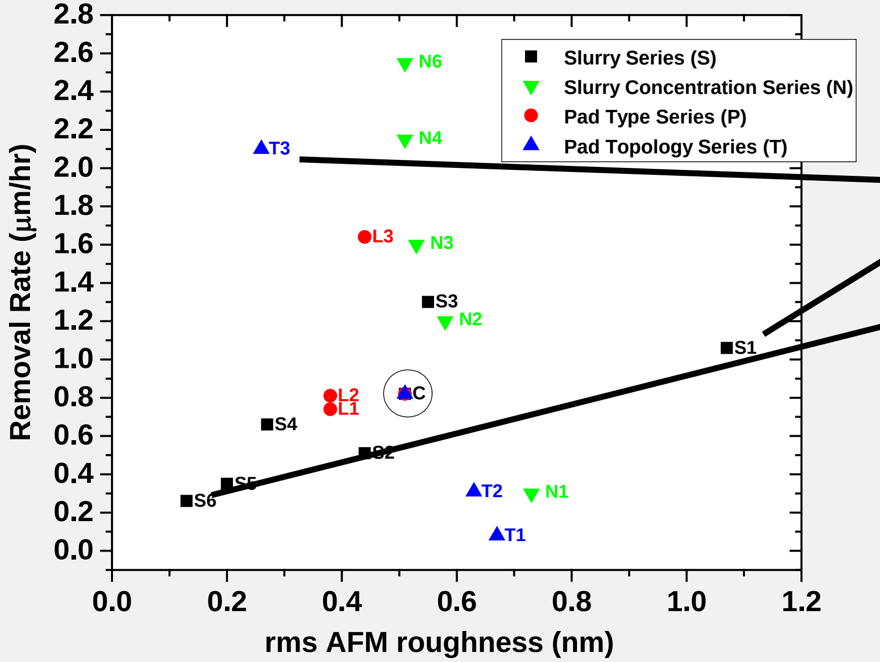
- Removal occurs over two regimes during polishing (molecular and plastic)
- Fused silica and BK7 have similar removal functions
- Removal function for phosphate glass is higher
- Combining removal function with load/particle distribution allows for predicting roughness

Schematic Model of the parameters that affect roughness during polishing



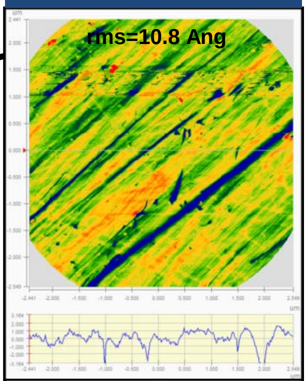
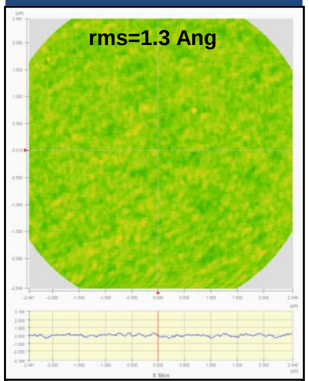
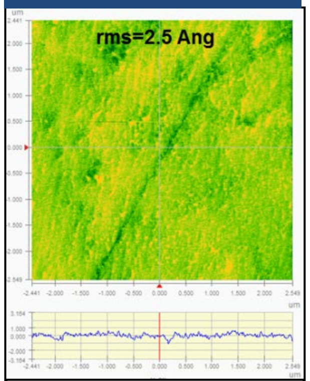
Key polishing variables were measured to test and validate the EHMG polishing model

Plot of measured removal rate & roughness on Fused Silica



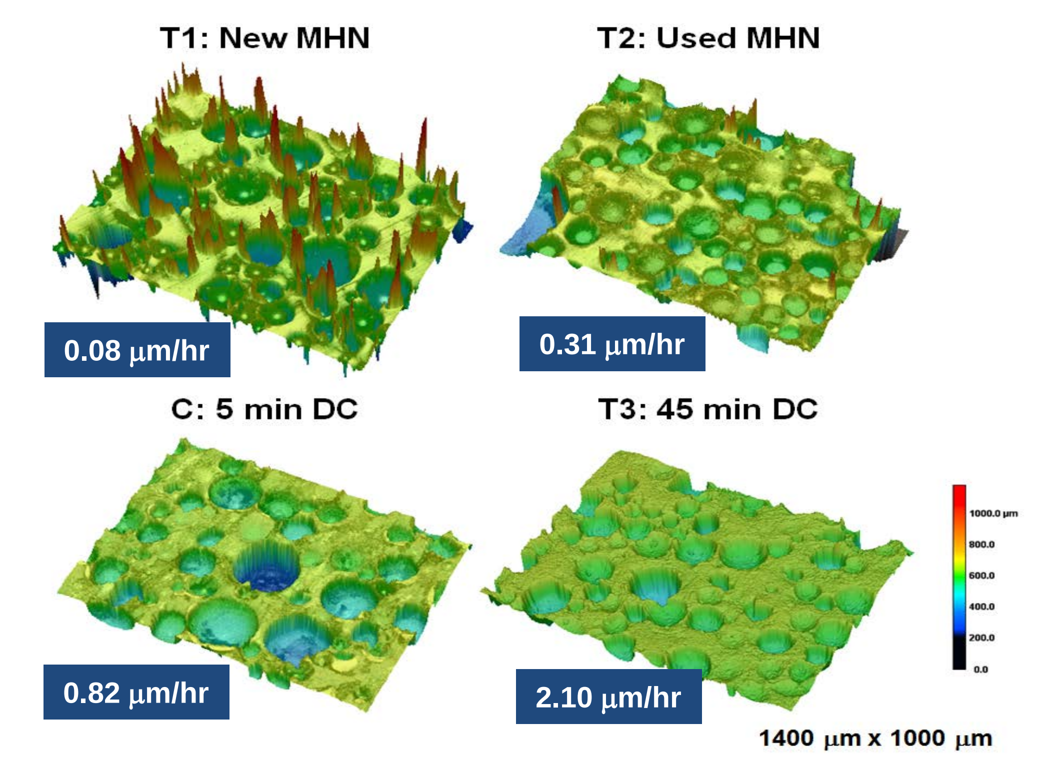
Key polishing variables:

1. Slurry PSD
2. Slurry Concentration
3. Pad Topology
4. Pad Type

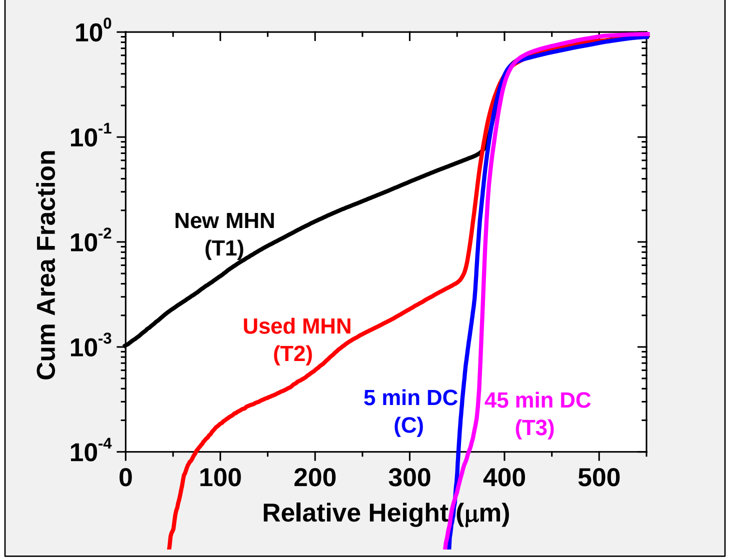
Wide PSD	Narrow PSD	Flat Pad
		
High roughness High removal rate	Low roughness, Low removal rate	Low roughness High removal rate

Pad topology during polishing strongly influences removal rate

MHN Pad Surface Topology (Confocal Microscope Images) with various surface treatments



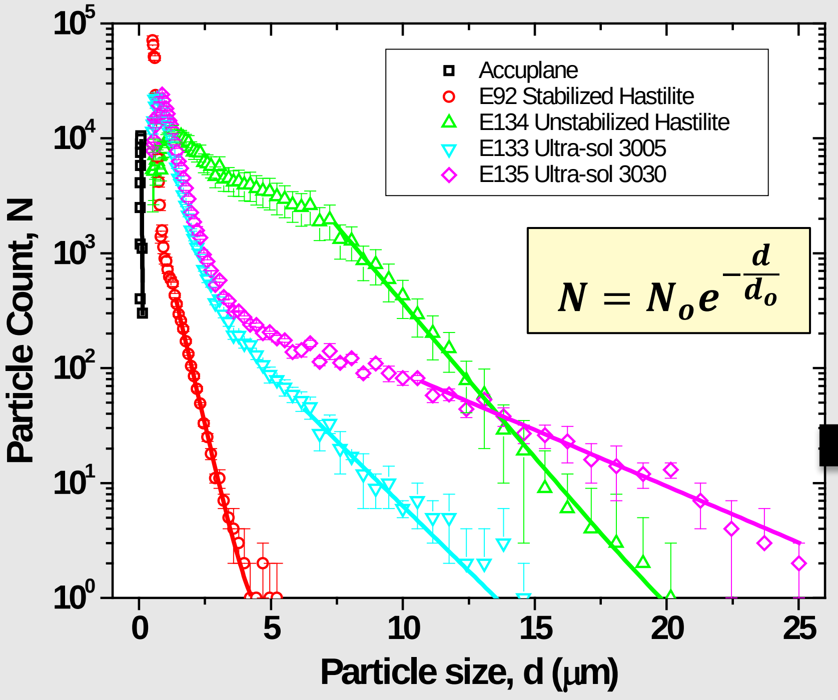
Pad Height Histograms



- **Tall pad asperities (100's μm) are removed with diamond conditioning pad treatment**
- **Removal rate increased from 0.08 μm/hr to 2.10 μm/hr; 26x increase**

Slurry's PSD* strongly correlates with workpiece roughness and removal rate

Measured PSD of ceria slurries

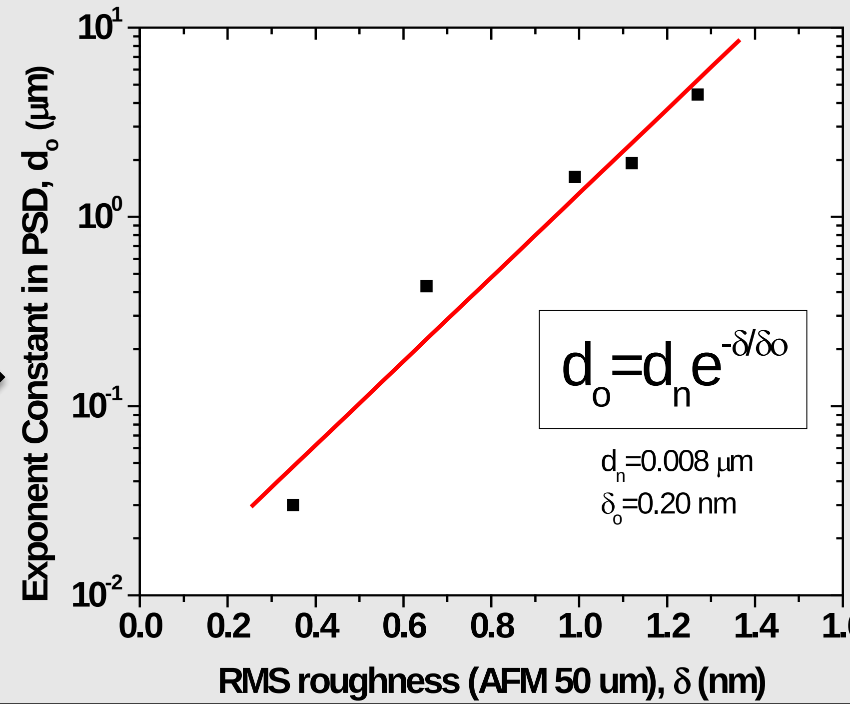


The tail end of each slurry follows a single exponential distribution

T. Suratwala et. al., *J. Am. Cer. Soc* 97(1) (2014) 81

*Particle size distribution

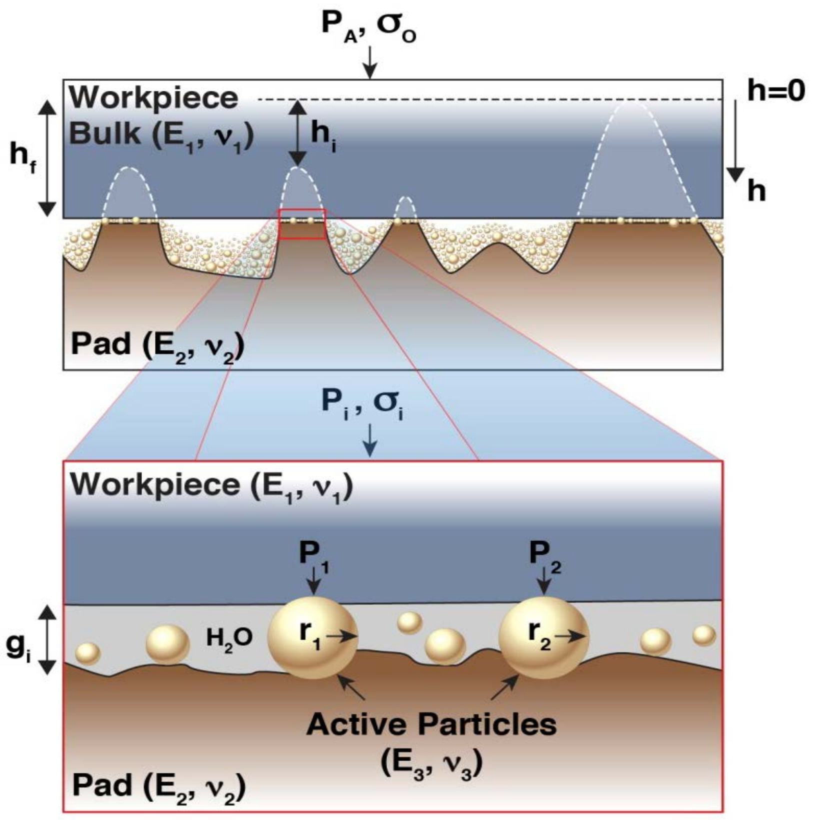
Exponent constant in PSD of slurry vs RMS roughness of polished surface



The slope of the slurry's PSD quantitatively scales with the rms roughness

EHMG (Esemble Hertzian Multi-Gap) polishing model accounts for both slurry PSD & pad topology to determine RR and roughness

EHMG Model Setup



Stress on each asperity (σ_i)

$$\sigma_i = \frac{h_f - h_i}{t_p} E_2$$

Gap for each asperity of stress (σ_i)

Load on each particle (Hertzian Contact)

$$P(r, g_i) = \frac{4}{3} \mathrm{E}_{eff} \sqrt{r(2r - g_i)^3}$$

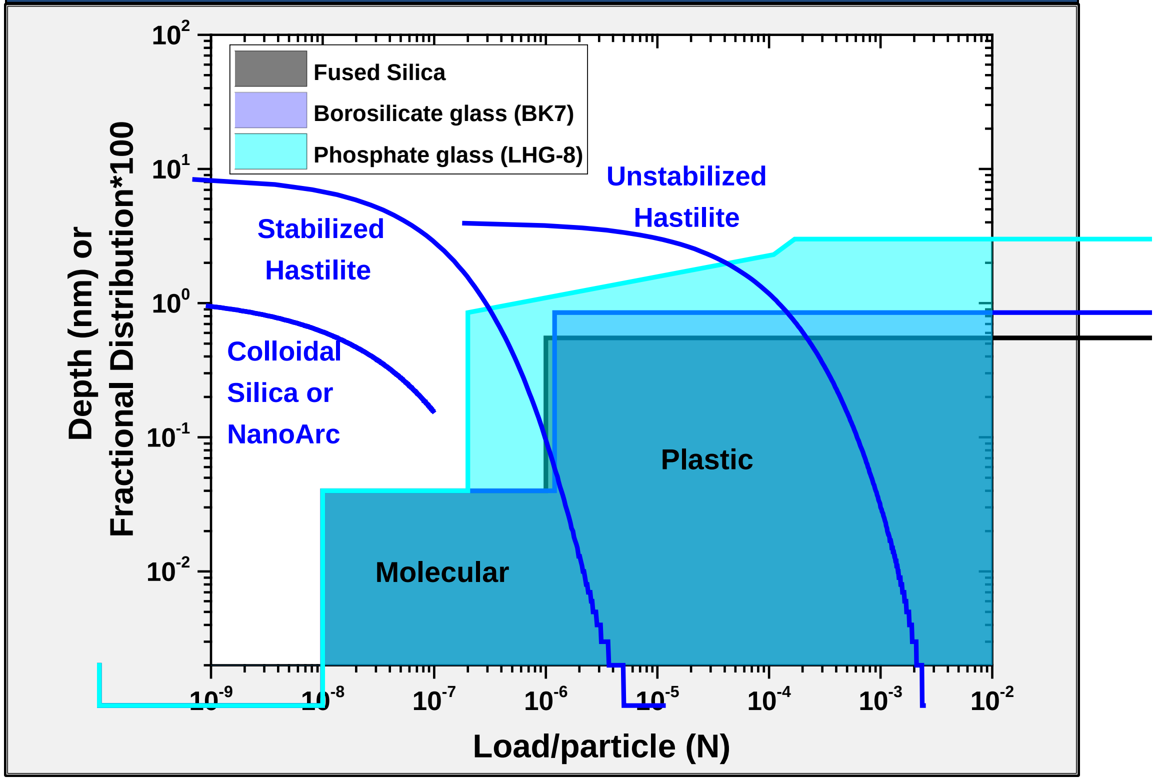
Load Balance on each asperity

$$\sigma_i = \mathrm{N}_b \int_{\frac{g_i}{2}}^{\infty} 2F(r)\, P(r, g_i)\, dr$$

Interface gap (g_i) for each asperity is determined by asperity stress (σ_i) and particle size distribution F(r) over whole workpiece

Load/particle distribution calculated using EHMG model, combined with measured removal function, gives the removal amount for each slurry particle

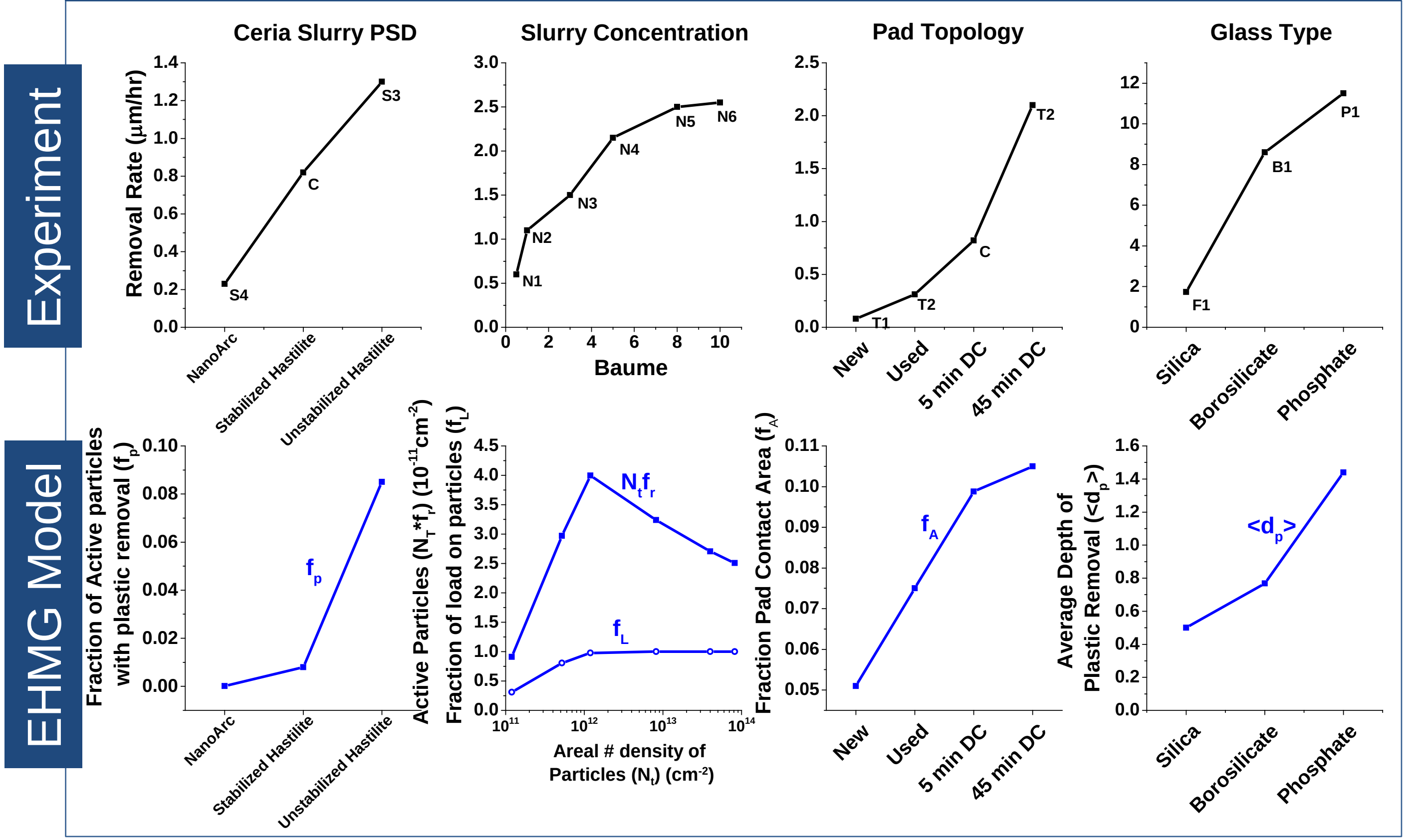
Single particle removal function & calculated load/particle distribution



This can be now used to calculate both removal rate and roughness during polishing

EHMG model compared with experiments expands our insight to the diverse factors affecting material removal rate

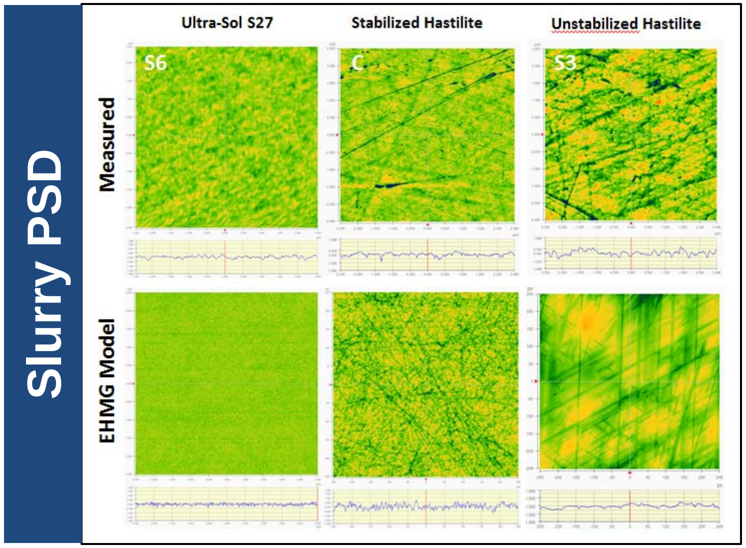
Measured removal rate & EHMG model Comparison

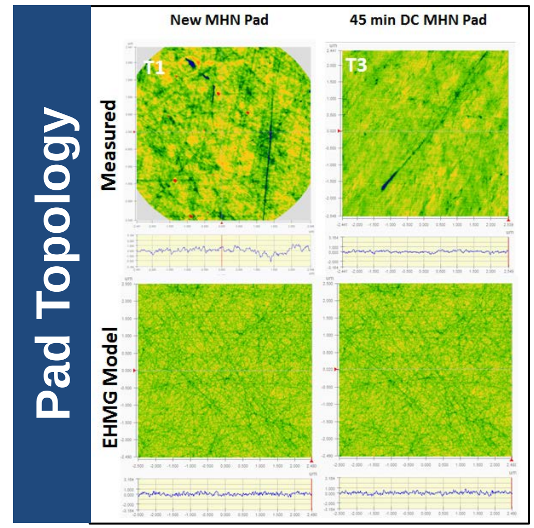


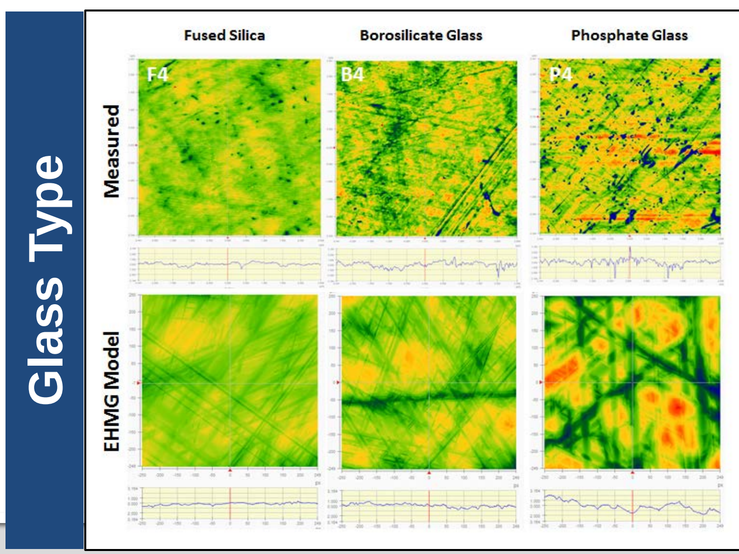
- Widening PSD increases load/particle & fraction of removal by plastic removal (f_p)
- Increasing slurry conc increases active particles density ($N_t f_r$) and fraction of load carried by particle (f_L)
- Increasing pad flatness increases fraction of pad area making contact (f_A)
- Change in glass type change removal depth by plastic removal (d_p)

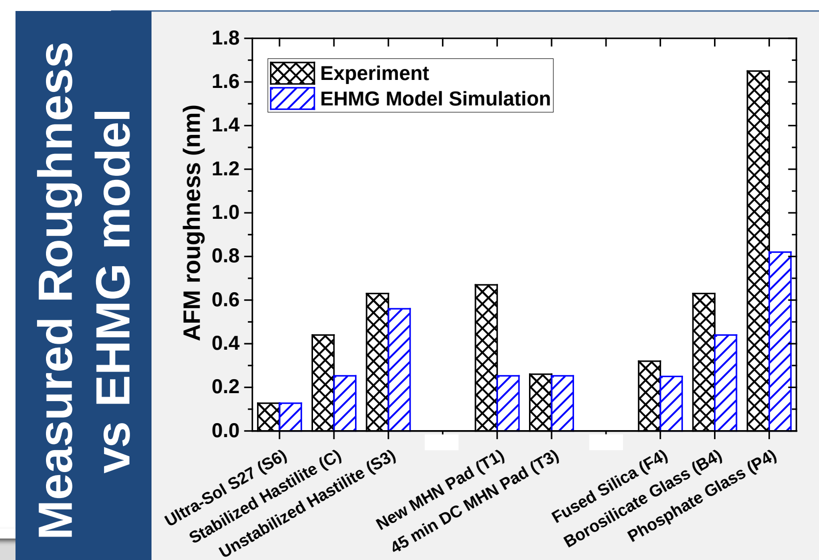
$$\frac{dh}{dt} \approx N_t\, f_A\, f_L\, f_r V_r \left(f_p \langle d_p \rangle \langle 2a_p \rangle + f_m \langle d_m \rangle \langle 2a_m \rangle \right)$$

EHMG model also simultaneously simulates trends in observed AFM roughness over a variety of polishing parameters









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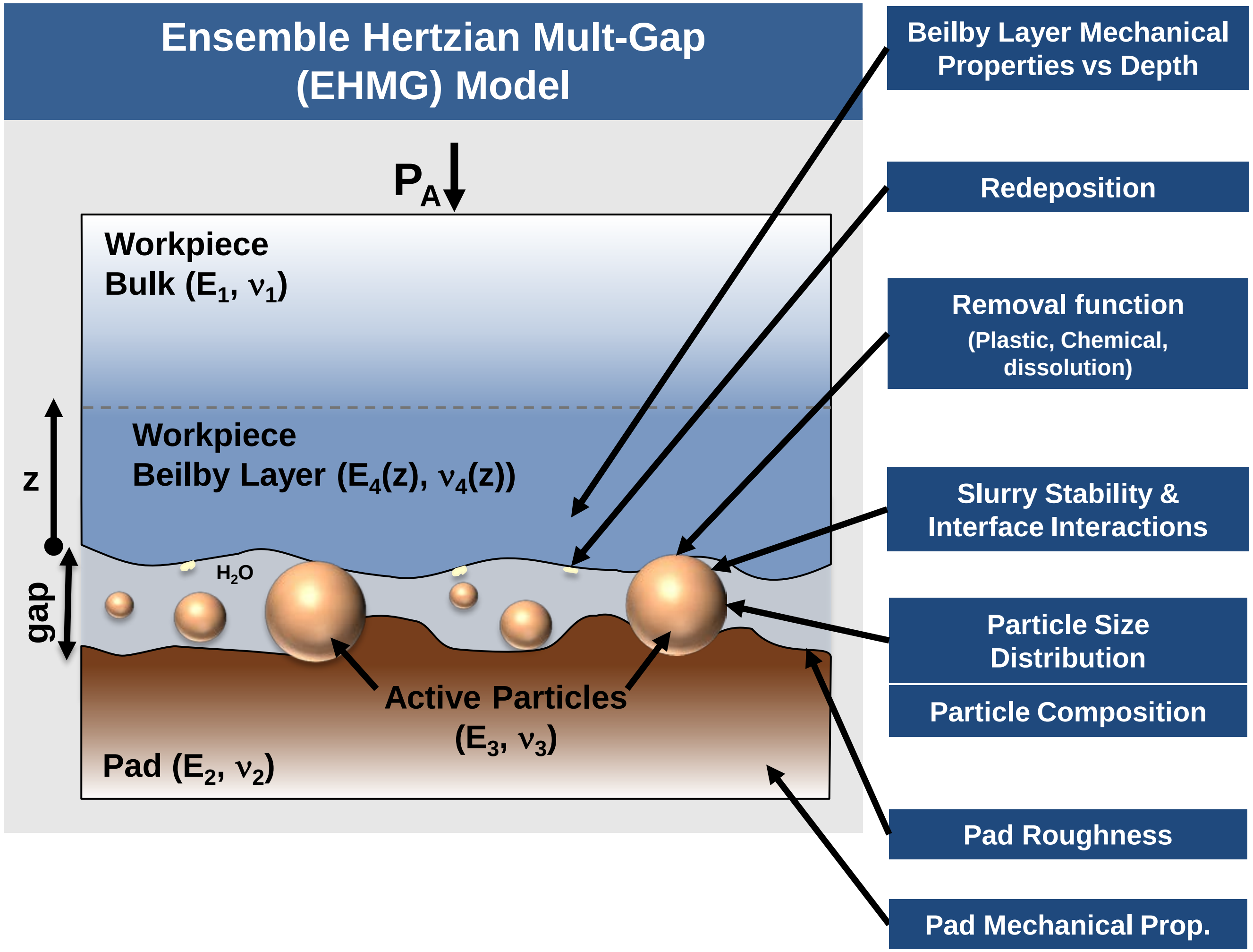

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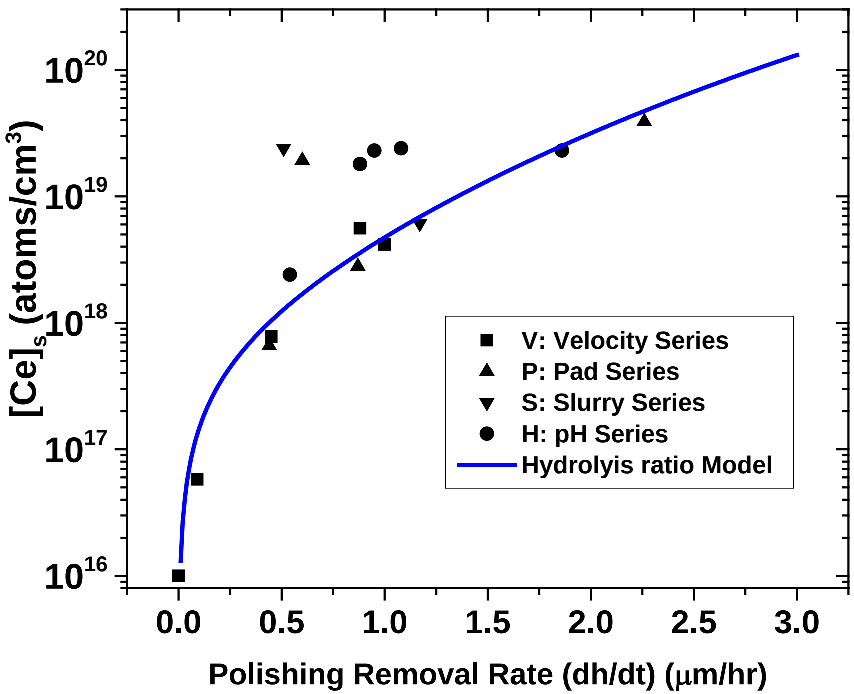
The ceria concentration variation with polishing rate can be predicted using temperature dependent hydrolysis ratio (r)

Measured vs Calculated $[Ce]_s$ using temperature dependent hydrolysis ratio



E=10 kcal/mole
r_o=80

$$C_o = \frac{S_p}{d} r(T) = \frac{S_p}{d} r_o e^{\frac{-E}{R\,T\left(\frac{dh}{dt}\right)}}$$

- **Proposed Mechanism**

  - As removal rate increase, there is an increase interface temperature

  - The resulting temperature rise causes a Arrhenius change to hydrolysis ratio (r) of Si-O and Ce-O

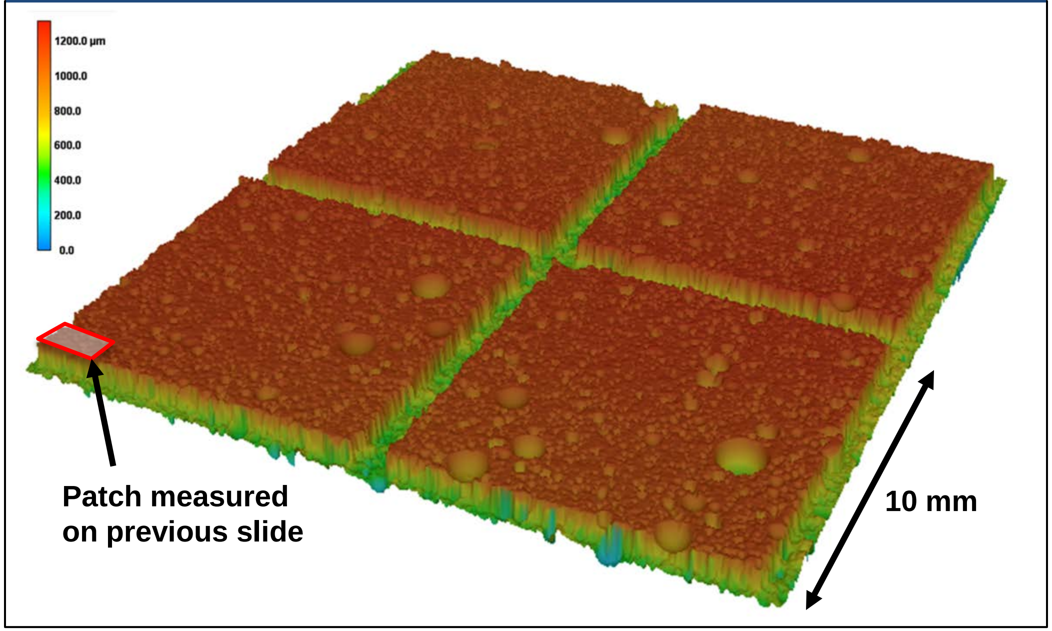
  - Increase in r results in more Ce deposition to the surface

- **Best fit activation energy (E) of 10 kcal/mole is consistent with literature values of Si-O-Si hydrolysis***

*Cypryk, Organometallics 21 (2002) 2165

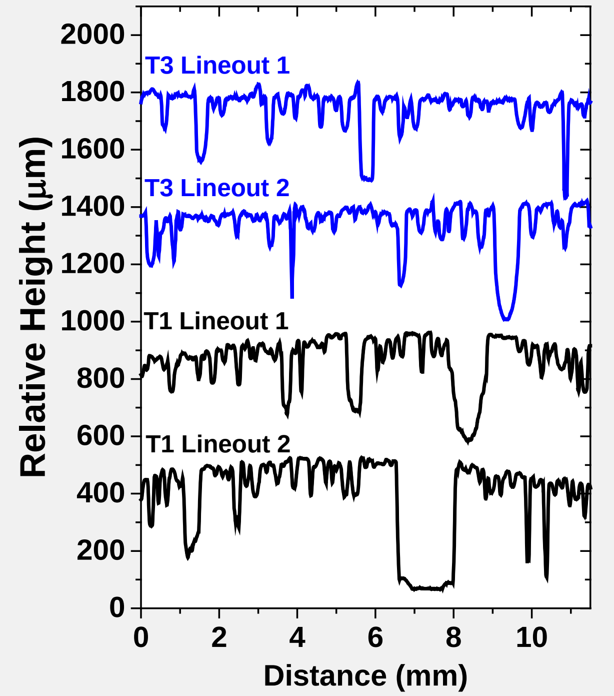
Aggressive DC treatment also flattened the pad over longer spatial scale lengths

Surface Topology for 45 min DC MHN pad (T3)



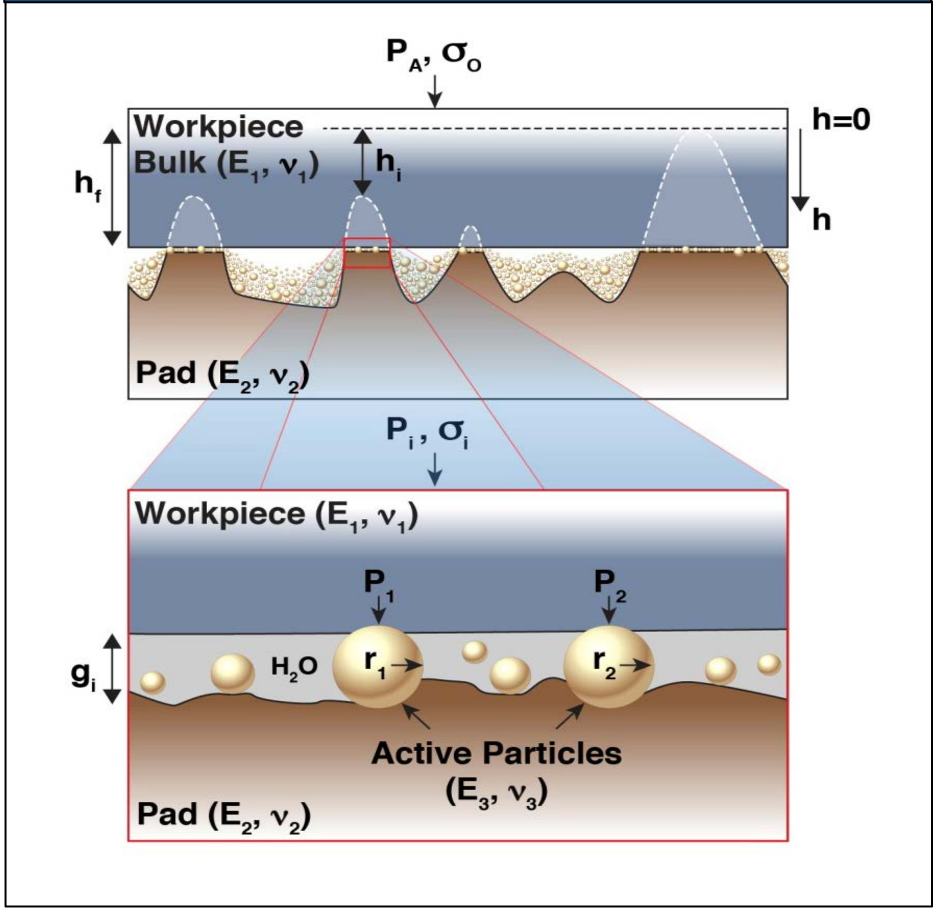
DC= diamond conditioning

**PAD Surface lineouts
with & without DC treatment**



EHMG (Esemble Hertzian Multi-Gap) polishing model accounts for both slurry PSD & pad topology to determine RR and roughness

EHMG Model Setup



T. Suratwala et. al., *J. Am. Cer. Soc* (2016) accepted

- <u>Key Inputs:</u> Slurry PSD & Pad Topology
- Using pad height histograms:
  - Pad asperities compress leading to single value gap of pad (g_p) based on load balance
  - Fraction of pad area making contact is calculated
- Each asperity compresses by height (h_i) resulting in stress (σ_i)
- Using slurry PSD at <u>each asperity</u> land–workpiece interface, slurry particles are loaded with a unique gap (g_i) following load balance
- Load/particle distribution is calculated from summing all pad asperities

The EHMG polishing model is used to expand key parameters affecting material removal rate

Macroscopic Preston's Equation

$$\frac{dh}{dt} = k_p \sigma_o V_r$$

Preston's Constant | Applied Pressure | Relative Velocity

Revised Microscopic Level Preston's Equation from EHMG model

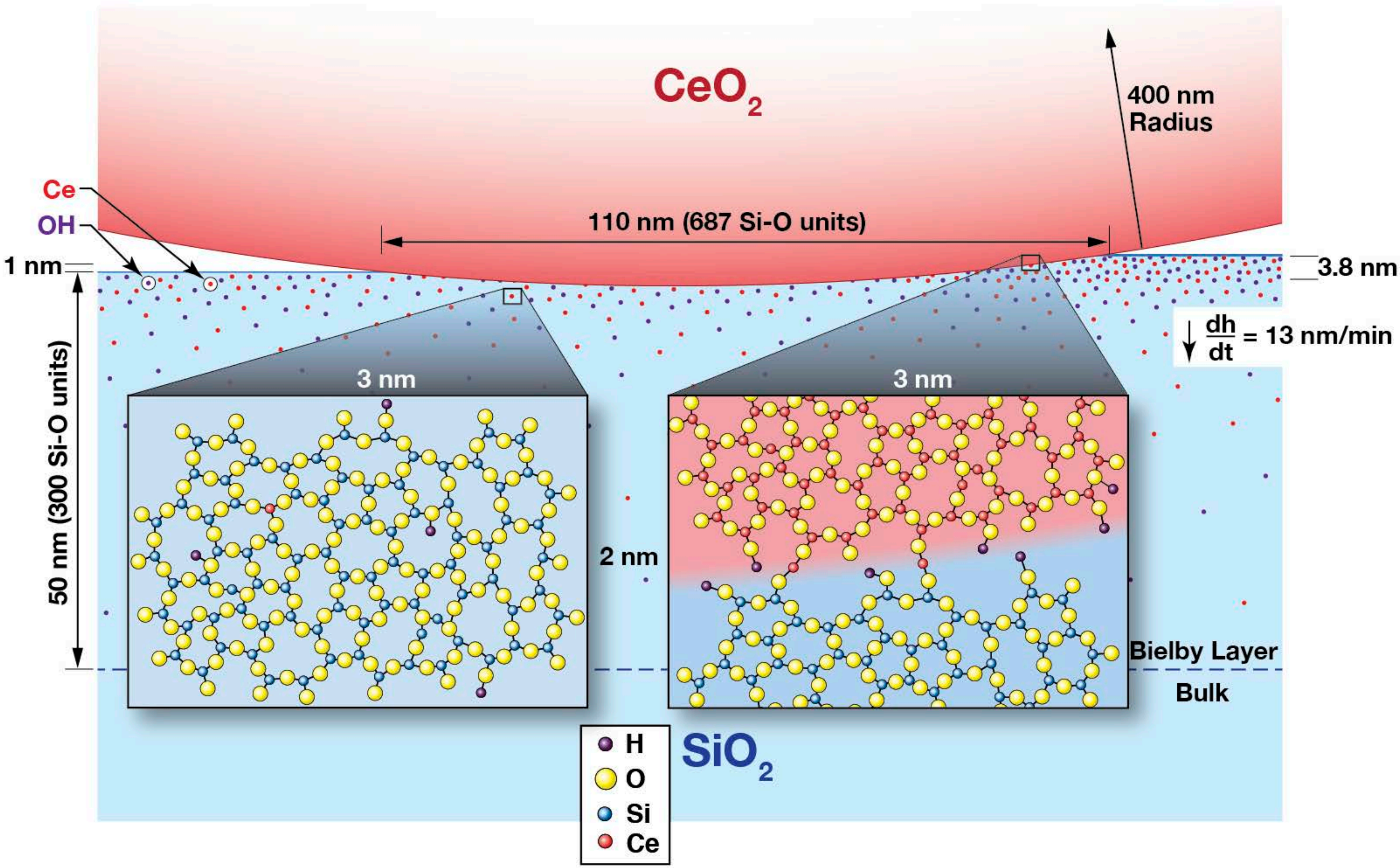
Summation of removal for each particle either by molecular removal or by plastic removal

Plastic Removal | Molecular Removal

$$\frac{dh}{dt} \approx N_t \, f_A \, f_L \, f_r V_r \left(f_p \langle d_p \rangle \langle 2a_p \rangle + f_m \langle d_m \rangle \langle 2a_m \rangle \right)$$

- Particle # Density (#/area)
- Fraction of pad area making contact
- Fraction of applied load on particles
- Fraction Active particles
- Fraction removal by molecular mechanism
- Removal depth
- Contact diameter

Combining all these measurements, a structural polishing model at particle-workpiece interface has been proposed



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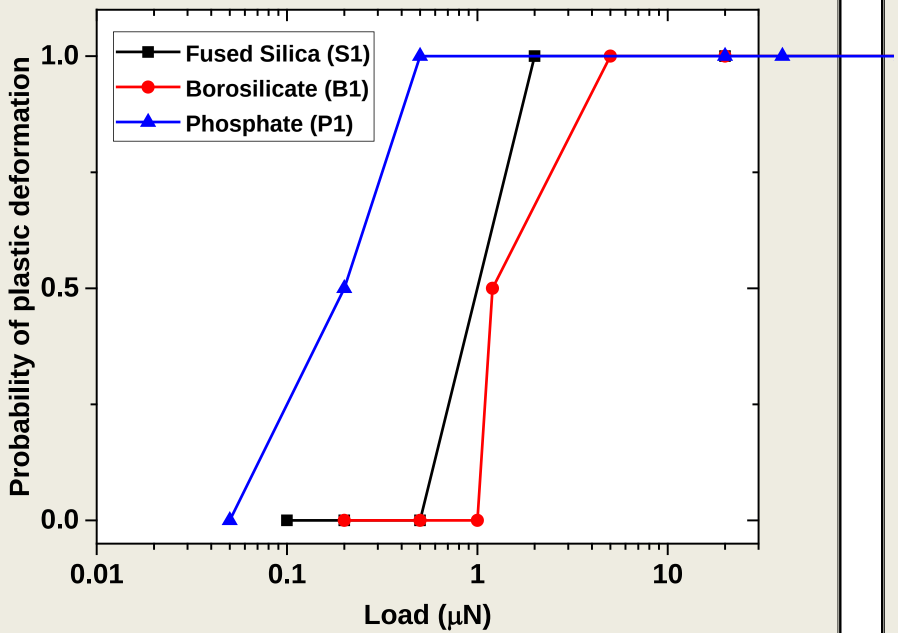
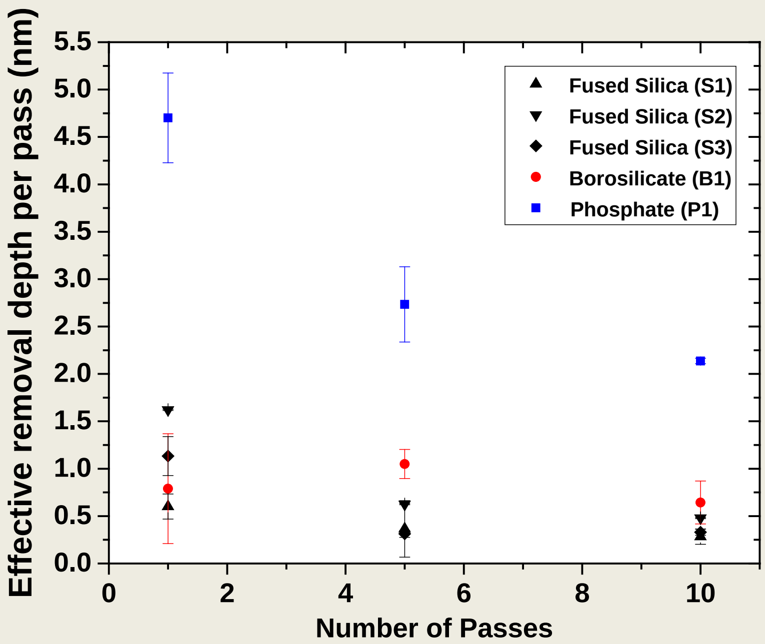
Both initiation load and multi-scratching at same location using AFM tip reveal insight to the fundamental removal function during ‘plastic’ type polishing

Determination of critical load for plastic deformation



Effect of multi-scratch on scratch depth at 110 μN on various glasses



Comparison of nanoscratching on different materials (function of # of passes & environment)